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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:

ATSUSHI SHIOTA ET AL.

: GROUP ART UNIT: 1712

SERIAL NO: 09/770,289

: EXAMINER: FEELY, M.

FILED: JANUARY 29, 2001

FOR: PROCESS FOR PRODUCING
SILICA-BASED FILM, SILICA-
BASED FILM, INSULATING FILM,
AND SEMICONDUCTOR DEVICE

DECLARATION UNDER 37 C.F.R. § 1.131

COMMISSIONER FOR PATENTS
ALEXANDRIA, VA 22313-1450

SIR:

Now come Atsushi SHIOTA and Kouji SUMIYA, who declare and state that:

1. We are the co-inventors of the above-identified application.
2. Prior to June 11, 1999, we reduced to practice the process for producing a silica-based film claimed in the above-identified application.

3. This reduction to practice is supported by the attached Exhibits A-E.

4. Exhibits A-C are from a laboratory notebook recorded by Atsushi SHIOTA.

Exhibit A is the notebook cover showing that the notebook contains records of "EB / Cure Experiment", i.e., experiments using electron beams for curing. Exhibit B is a record of electron beam exposures of "Siloxane" at doses of "3000-5000 μ C/cm²". Exhibit C is a

5. Exhibit D shows the dielectric constant ("k"), elastic modulus ("E"), universal hardness ("H") and thickness ("t") of siloxane samples cured with electron doses of 10, 50, 100, 500, 1000 and 3000 $\mu\text{C}/\text{cm}^2$. As discussed above, "LDK-5105" is the sample number assigned to a particular siloxane.

6. Exhibits A-D demonstrate electron beam exposure of siloxane films at doses in a range of from 1 to 200 $\mu\text{C}/\text{cm}^2$ results in films having a dielectric constant of 3 or lower.

7. The feature of "silicon carbide bonds represented by Si-C-Si" is inherent in siloxane films electron beam irradiated at a dose in the range of from 1 to 200 $\mu\text{C}/\text{cm}^2$. The Si-C-Si bonds appear in infrared spectra as a peak at 890 cm^{-1} . Specification at page 27, line 26 to page 28, line 2.

8. Exhibit E is an infrared spectrum of a siloxane film that has received an electron dose in the range of from 1 to 200 $\mu\text{C}/\text{cm}^2$.

9. We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

10. Further deponents saith not.

Date: 27 Aug 2003

Atsushi Shiota
Atsushi SHIOTA

Date: 27 Aug 2003

Kouji Sumiya
Kouji SUMIYA

Attachments: Exhibits A-E

FOR CLEAN ROOM USE

NEW STACLEAN
Note Book

TEL / Name / E.P. / Class / Experiment

JSR 磯田

25-49 Chabbin Pan

0461 328

Test time 6. 50

7

50

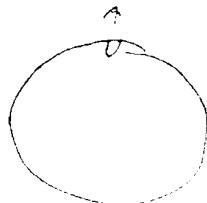
Samples 51 52 53 54 55 56 57 58
Si/S-N 50

Siloxane 506, 3,000 - 5,000 μ C/cm²

4. ~ 6 min

At 5

N & K



Stage 1 8 mm

1. Vacume
2. Move Stage

Edita Tejnik

3 Task / Baseline Scan

Library / Default. Bish Street

Ling. S-3447.

	Dose	Init	n	6
4CD-5105/9C1802		6550A ^{0.9}	[1.266]	
14SJF/	10	6388.5	40.20	1.260 (0.000E)
16SJG/2	80	6006.7	109.74	1.260 (0.000E)
15SJG/0	100	5778.2	128.16	1.2812 (0.00462)
11SJH/1	500	4840.9	170.06	1.3005 (0.00930)
12SJEO	1,000	4378.5	285.35	1.358 (0.0665)

Energ 5 KeV

Dose 10 uC/ 50, 100, 1000 uC/a

Current 5 mA

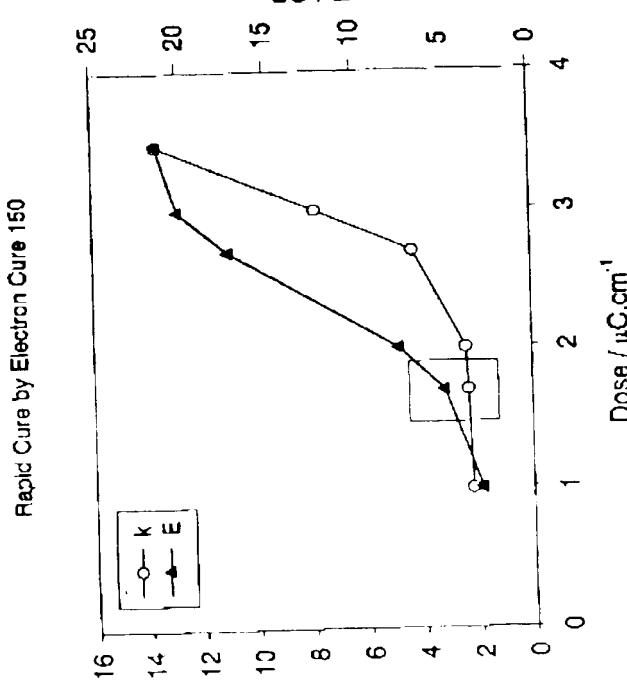
Temp. 350 °C

Gas Ar

Pressure 10 mTorr

IP6.676 CMF

Results Of Rapid Cure with EB



Operation Condition.
Acc. Vol. : 5 keV
Current : 5 mA
Pressure : 10 mTorr
Flow Gas : Ar
Temp. : 400 C

It was possible to cure rapidly (< 5 min.) using EB.
Optimum dose to increase Young's moduli without increasing k value was ca. 50 mC/cm^2 .
Moisture adsorption, thermal volatile should be determined.

